

# Experiment 6

## Synthesis and Analysis of a Complex Iron Compound

Part Deux: Oxalate Content Analysis by Redox Titration

CH 204 Spring 2006

Dr. Brian Anderson

# But first...

Last week:

Synthesis

Metal complex coordination compounds

Oxidation/Reduction (Redox) reactions

Calculating theoretical yield and percent yield

# Review of Redox Chemistry

Oxidation: Loss of electrons. Oxidation number increases (gets more positive).

Reduction - Gain of electrons. Oxidation number decreases (moves more negative).

Redox — A chemical reaction in which some atoms are oxidized and others are reduced. Individual atoms within compounds are oxidized or reduced.

# What is an oxidation number?

A method for keeping track of the movement of electrons in redox reactions. It's like treating all compounds as if they're ionic compounds, even when we know they're not.

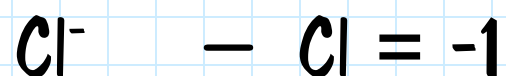
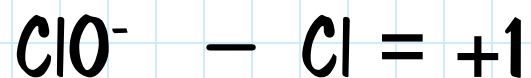
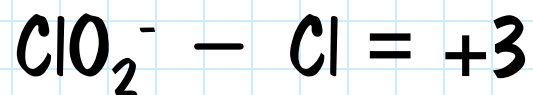
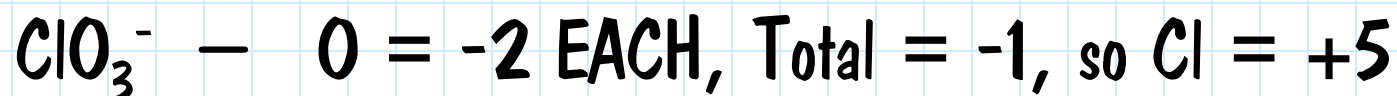
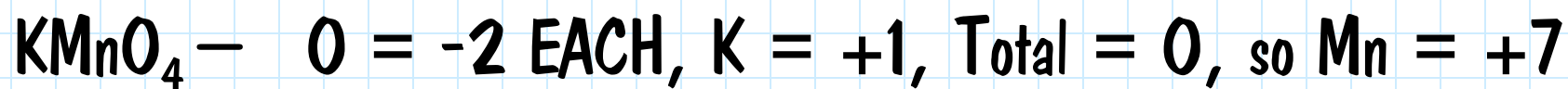
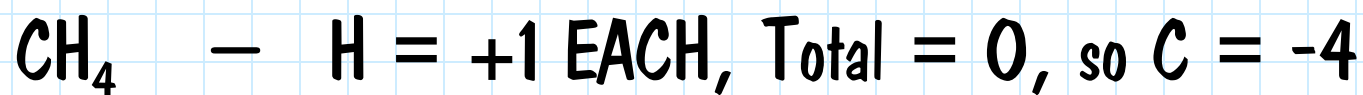
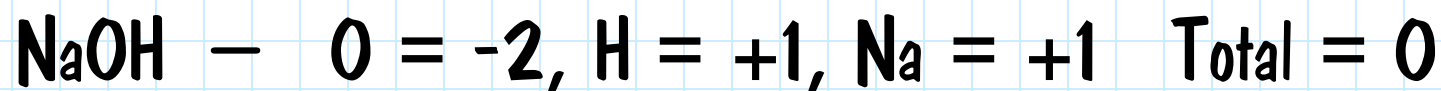
Each individual atom is assigned an oxidation number according to a few simple rules:

- Atoms in their elemental state have an oxidation number of zero.
- Monatomic ions have an oxidation state equal to the charge on the ion.
- All the oxidation states in a neutral molecule add up to zero.
- All the oxidation states in an ion add up to the charge on the ion.

# Common Oxidation States

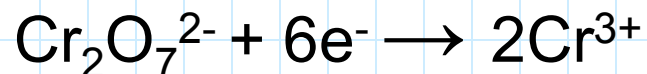
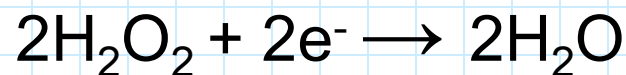
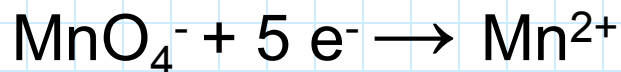
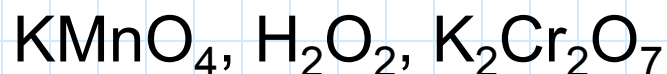
- Hydrogen is almost always  $+1$ .
  - 0 in elemental hydrogen ( $H_2$  gas)
  - $-1$  in metal hydrides such as  $LiAlH_4$
  
- Oxygen is almost always  $-2$ .
  - 0 in elemental oxygen ( $O_2$  gas)
  - $-1$  in peroxides such as  $H-O-O-H$

# Some examples



# Oxidizing Agents

An oxidizing agent is something that oxidizes other chemicals -- and therefore gets reduced itself. Strong oxidizing agents often have lots of oxygen atoms in them. Common examples include



These are **NASTY!** Nasty nasty nasty!

# Reducing Agents

A reducing agent reduces other compounds and therefore gets oxidized itself. Hydrides are commonly used reducing agents, particularly Lithium Aluminum Hydride ( $\text{LiAlH}_4$ ) and Sodium Borohydride ( $\text{NaBH}_4$ ).



# But wait, there's more!

Those were some common strong oxidizing and reducing agents, but under the right circumstances,

- Anything that can be reduced can act as an oxidizing agent
- Anything that can be oxidized can act as a reducing agent

# Our redox reaction

We will use  $\text{MnO}_4^-$  to oxidize the oxalate ligands surrounding the  $\text{Fe}^{3+}$  from  $\text{C}_2\text{O}_4^{2-}$  to  $\text{CO}_2$ .

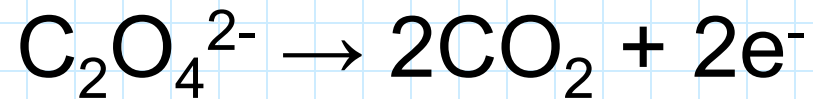
Last week we oxidized  $\text{Fe}^{2+}$  to  $\text{Fe}^{3+}$  using hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) as the oxidizing agent.

Peroxide is not a strong enough oxidizer to oxidize the oxalate to  $\text{CO}_2$ , but permanganate is.

# Oxidation half-reaction

Oxidation of  $\text{C}_2\text{O}_4^{2-}$  to  $\text{CO}_2$  is simple enough:

(Remember, half-reactions do not include the other reactant)

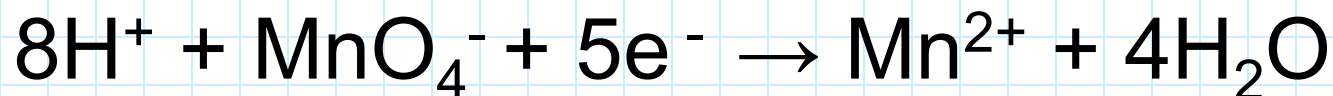


# Reduction half-reaction

The oxidizing agent,  $\text{MnO}_4^-$ , gets reduced to  $\text{Mn}^{2+}$

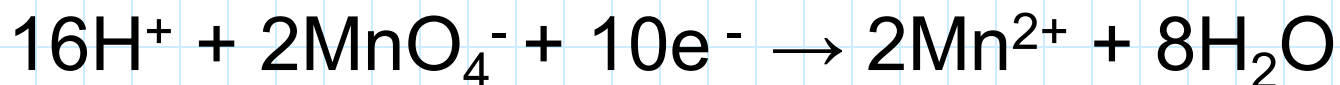
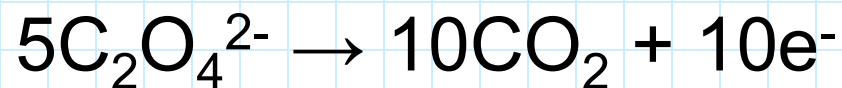
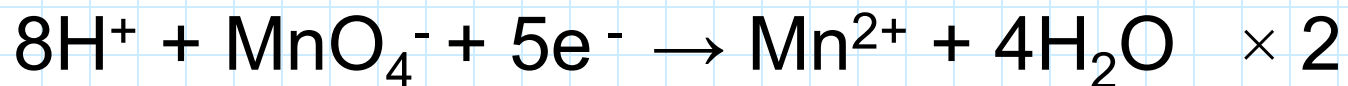
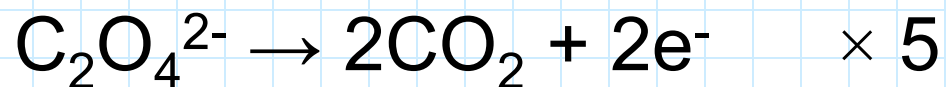
$\text{MnO}_4^- + 5e^- \rightarrow \text{Mn}^{2+} + \text{a whole bunch of oxygens}$

In acidic solution,

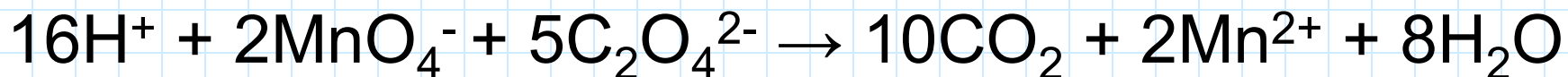


# Add the two half reactions

First multiply the equations in order to balance out the electrons:



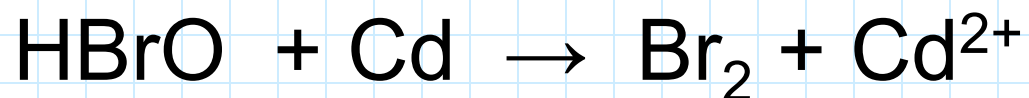
The equation for the overall reaction is:



# Balancing redox reactions

- Identify which species is being oxidized and which one is being reduced.
- Separate them into half reactions (usually one of the half reactions is trivial to balance).
- Balance the main atom.
- Add  $\text{H}_2\text{O}$  to balance O, then add  $\text{H}^+$  to balance H.
- Balance the charge using electrons.
- Add the two half-reactions (cancel out electrons)
- If the reaction takes place in basic solution add  $\text{OH}^-$  to both sides to turn  $\text{H}^+$  into  $\text{H}_2\text{O}$ , and then cross out redundant waters.

# Balance this redox reaction



# Always balance in acidic solution

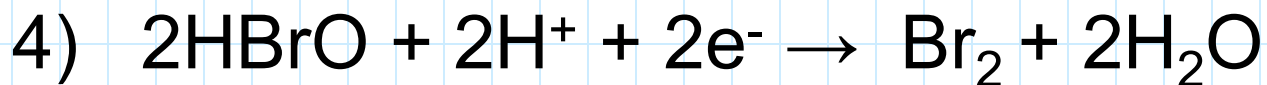
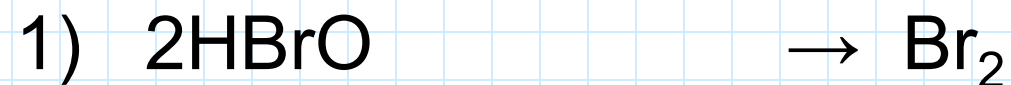
As easy as 1-2-4.

1) Balance the oxidized/reduced atoms

2) Balance oxygens using  $\text{H}_2\text{O}$

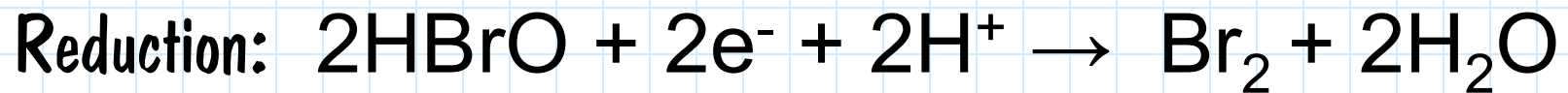
3) Balance hydrogens using  $\text{H}^+$

4) Balance charge using  $e^-$

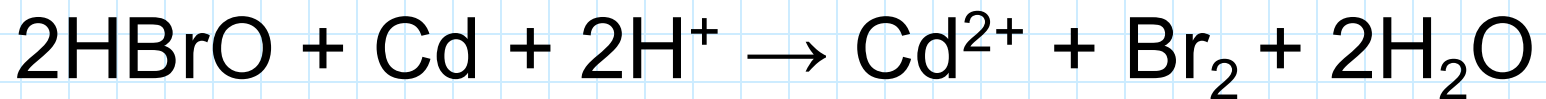




# Add the two half-reactions



Overall Equation:



# What if the solution is basic?

Here's what Whitten, Davis, Peck, and Stanley say:

To balance in a basic solution, for each O needed

(1) Add *two*  $\text{OH}^-$  to the side needing O

and

(2) Add *one*  $\text{H}_2\text{O}$  to the other side

Then for each H needed,

(1) Add *one*  $\text{H}_2\text{O}$  to the side needing H

(2) Add *one*  $\text{OH}^-$  to the other side.

# Here's what the new book suggests

"In basic solution, balance O by using  $\text{H}_2\text{O}$ ; then balance H by adding  $\text{H}_2\text{O}$  to the side of each half reaction that needs H and adding  $\text{OH}^-$  to the other side."

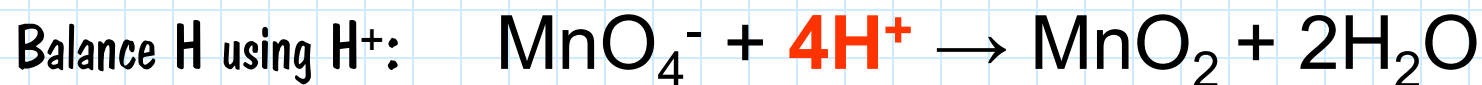
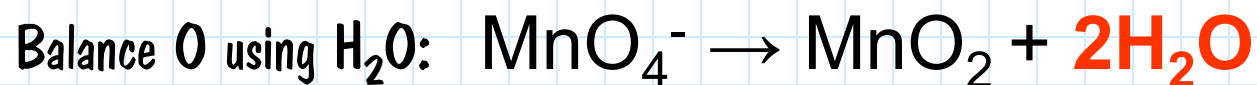
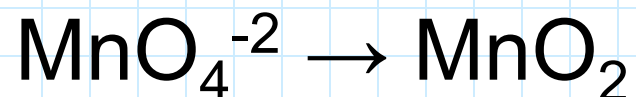
"When we add  $\dots \text{OH}^- \dots \rightarrow \dots \text{H}_2\text{O} \dots$  to a half-reaction, we are effectively adding one H atom to the right. When we add  $\dots \text{H}_2\text{O} \dots \rightarrow \dots \text{OH}^- \dots$  We are effectively adding one H atom to the left. Note that one  $\text{H}_2\text{O}$  molecule is added for each H atom needed."

# Do it the E-Z way instead

Balance the equation in acidic solution,  
and if it's supposed to be in basic solution,  
just add enough  $\text{OH}^-$  to both sides  
to get rid of all the  $\text{H}^+$ .

Just like this...

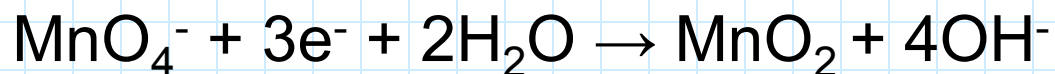
Permanagante is reduced to manganese (IV) oxide in basic solution:



Add one  $\text{OH}^-$  for every  $\text{H}^+$ . Add  $\text{OH}^-$  to both sides!



Combine waters and delete redundant waters:



# Balancing redox reactions review

- Separate the reactants into half reactions (usually one of the half reactions is trivial to balance).
- Balance the main atom.
- Balance the half-reactions using  $\text{H}_2\text{O}$  to balance O, then use  $\text{H}^+$  to balance H. Balance the charge with electrons.
- Add the two half-reactions — electrons must cancel.
- If necessary, convert acidic solution to basic by adding  $\text{OH}^-$  to both sides and crossing out spectator water molecules.

# Today: Sample prep and three titrations

**Land mine!** 1:1 mixture of ethanol/water means mix them together in a beaker **BEFORE** you pour them in!

The  $\text{KMnO}_4$  solution is already standardized and ready to go.  
Make sure you record the concentration!

Actual  $\text{KMnO}_4$  concentration is about **0.035 M**, not **0.02 M**.

Take **60 ml** instead of **80 ml**.